

LOW TEMPERATURE PLASMA CARBURIZING OF AISI 316L AUSTENITIC STAINLESS STEEL AND AISI F51 DUPLEX STAINLESS STEEL

CEMENTAÇÃO SOB PLASMA A BAIXA TEMPERATURA DO AÇO INOXIDÁVEL AUSTENÍTICO AISI 316L E DO AÇO INOXIDÁVEL DUPLEX AISI F51

Carlos Eduardo Pinedo

PhD, University of Mogi das Cruzes and Heat Tech Technology for Heat Treatment and Surface Engineering Ltd, Rod. Índio Tibiriçá 1555-B, ZIP 08620-000, Suzano, SP, Brazil.

André Paulo Tschiptschin

PhD, Professor, Metallurgical and Materials Engineering Department, University of São Paulo, Av. Prof. Mello Moraes 2463, ZIP 05508-030, São Paulo, SP, Brazil

Abstract

In this work an austenitic AISI 316L and a duplex AISI F51 (EN 1.4462) stainless steels were DC-Plasma carburized at 480°C, using CH₄ as carbon carrier gas. For the austenitic AISI 316L stainless steel low temperature plasma carburizing induces a strong carbon supersaturation in the austenitic lattice and the formation of carbon expanded austenite (γ_C). For the duplex stainless steel AISI F51 the austenitic grains are also transformed to carbon expanded austenite (γ_C) while ferritic grains are first transformed to austenite, due to carbon diffusion and then to carbon expanded austenite (γ_C). For both steels the austenite lattice expansion induces high compressive stresses in the carburized layer and a strong surface hardening up to 1400HV.

Key-words: plasma carburizing, expanded austenite, hardening.

Resumo

Neste trabalho os aços inoxidáveis; austenítico AISI 316L e duplex AISI F51 foram cementados sob plasma a 480°C utilizando CH₄ como gás condutor de carbono. No aço AISI 316L a cementação conduz a uma supersaturação da austenita em carbono que leva à formação da austenita expandida (γ_C). No aço duplex AISI F51, na cementação, a fração austenítica também se transforma em austenita expandida (γ_C) e a fração ferrítica, com a supersaturação em carbono, transforma-se inicialmente para austenita e em seqüência para austenita expandida. Para os dois aços, a austenita expandida é uma fase que gera um elevado estado de tensões residuais de compressão promovendo um intenso endurecimento superficial, atingindo níveis superiores a 1400HV.

Palavras-chave: cementação sob plasma, austenita expandida, endurecimento.

1. Introduction

Austenitic and duplex stainless steels are widely used in a variety of applications within the chemical, refining and petrochemical industry, where high corrosion resistance and mechanical properties are required. However, the surface properties of these materials may be improved in order to reach a better performance in highly stressed tribological systems. Carbon steels have been surface hardened by nitriding diffusion processes for decades. For these steels case hardening is a consequence of nitride compound layer formation and of nitrides precipitation in the diffusion zone, namely γ' -Fe₄N and ϵ -Fe₂₋₃N (Cavaliere, et al, 2009). On the other hand, case hardening of austenitic stainless steels (ASS) can be achieved by intensive precipitation of chromium nitrides (CrN, Cr₂N) in the diffusion zone during nitriding at temperatures around 500°C, increasing hardness up to 1400 HV, but decreasing the corrosion resistance (Renevier, et al, 1999, Czerwicz, et al, 2000, Liang, et al, 2000). For low temperature nitriding, close to 400°C, the hardening mechanism changes from nitride precipitation to lattice distortion of the FCC austenitic phase, leading to formation of expanded austenite, with hardness close to 1400 HV and no loss of corrosion resistance (Fewell, et al, 2000, Borgioli, et al, 2005, Mingolo, et al, 2006). The hardening mechanism is related to high compressive residual stresses arisen from lattice distortion.

Plasma processing of stainless steels is the most suitable case hardening process because it avoids the need of a pre-removal step of the passive Cr₂O₃ layer by chemical or mechanical operations. During plasma processing the passive layer is removed by sputtering prior to the nitriding step without any damage to the part's surface. Based on the nitrogen expanded austenite concept, low temperature carburizing has been studied for austenitic and other stainless steels types (Sun; et al, 1999, Cao; et al, 2003, Michal, et al, 2006, Ceschini, et al, 2008, Souza, et al, 2009). In such treatments a hard and rather ductile layer increases wear properties and fatigue resistance without impairing the corrosion resistance and even increasing it in some cases. The aim of this work is to present results of low temperature plasma carburizing of an AISI 316L austenitic and an AISI F51 duplex stainless steel. Changes in the surface microstructure, as well as its influence on hardening, corrosion and wear resistance are presented.

2. Experimental procedure

Annealed round bars of commercial austenitic AISI 316L (ASS) and duplex AISI F51 (DSS) stainless steels were sliced into 3 mm thick specimens, mechanically ground and polished with diamond paste down to 1 μ m, prior to plasma carburizing. The chemical compositions of both steels are shown in Table 1.

Table 1 - Chemical composition. mass %.

	C	Si	Mn	Cr	Ni	Mo	N
AISI 316L	0.015	0.33	1.82	17.37	14.51	2.82	0.075
AISI F51	0.030	0.69	1.02	21.16	6.55	2.76	0.147

Low temperature plasma carburizing (LTPC) was carried out in a pulsed plasma reactor with a hot wall chamber. Passive film was removed by a sputtering step conducted at 400°C for 1 hour using high intensity pure hydrogen plasma. Carburizing was conducted at 480°C, during 12 hours. A gas mixture composition of 18(l/h)H₂:6(l/h)Ar:180(cl/h)CH₄ was used. During the treatment the carburizing temperature was measured by two thermocouples embedded in the samples.

The phases formed in the carburized layer were identified by XRD in a Philips diffractometer using CuK_α radiation, $\lambda = 0.1542$ nm, with a conventional $\theta/2\theta$ Bragg-Brentano symmetric geometry. The surface hardness of the carburized samples was measured using a Shimadzu Vickers microhardness system with a 50 g load.

3. Results and discussion

3.1 LTPC AISI 316L Austenitic Stainless Steel

X-ray diffractograms of the AISI 316L ASS before and after plasma carburizing are shown in Figure 1. Compared to the untreated austenite, the diffraction peaks are broader and shifted to lower diffraction angles, characteristics of carbon expanded austenite (γ_C) similar to the nitrogen expanded austenite (γ_N) identified by other authors after plasma nitriding a 316L austenitic stainless steel (Mingolo et al, 2006). Very high compressive stresses develop in the FCC lattice due to carbon supersaturation increasing the austenite lattice parameter up to 0.369 nm. For comparison, the FCC lattice parameter of the untreated austenite was evaluated as $a = 0.359$ nm, and that available from JCPDS-ICDD® data files is $a = 0.360$ nm (ICDD, Goldschmidt, 1949). The relative expansion of austenite lattice parameter (γ_C) when compared to the untreated austenite ($\Delta a/a$) was calculated as 2.8%.

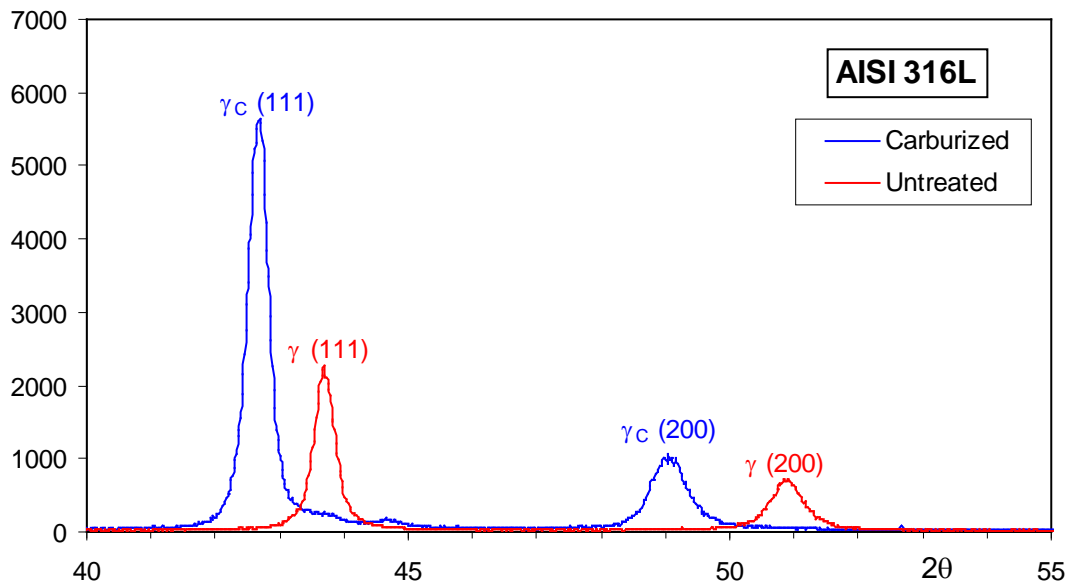


Figure 1 – XRD diffraction patterns for AISI 316L steel before and after carburization.

Figure 2 shows that the surface hardness after plasma carburizing AISI 316L ASS increased up to 1010 HV0.05. Compared to the hardness of the solubilized steel, 181 HV0.05, plasma carburizing increased hardness more than five times. Considering that ASS are not prone to be hardened by heat treatment, this surface treatment should potentially be used to increase tribological properties and service performance.

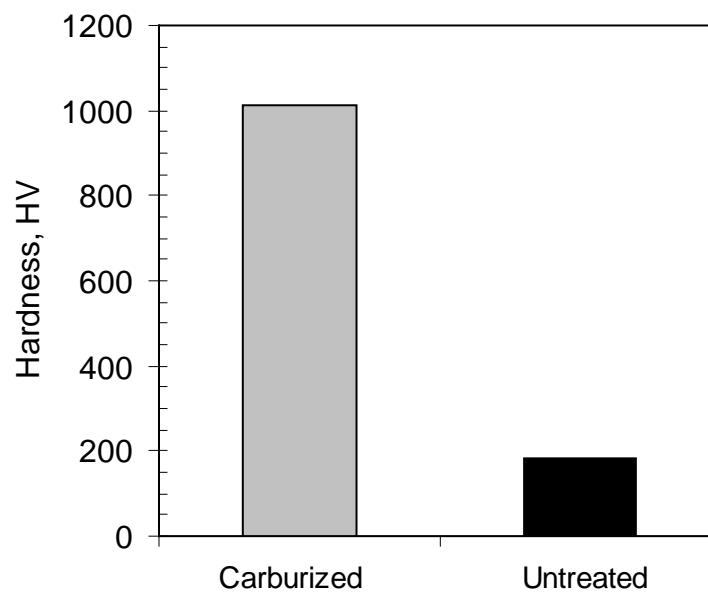


Figure 2 – Surface hardness after low temperature plasma carburizing AISI 316L ASS.

3.2 LTPC AISI F51 Duplex Stainless Steel

Figure 3 shows X-ray diffraction patterns for the solubilized and LTPC AISI F51 DSS. Before carburizing peaks referring to ferrite and austenite can be observed. The calculated lattice parameters for ferrite and austenite are 0.287 nm and 0.359 nm, respectively, close to those informed on ICDD files (ICDD, Goldschmidt, 194913, ICDD, Swanson, 1955) 0.286 nm and 0.360 nm. After carburizing the diffraction peaks are broader and shifted to lower diffraction angles, denoting lattice expansion. High intensity original peaks for ferrite (110) and austenite (111) disappear and a new high intensity and broader peak appears for $2\theta = 44.31^\circ$ indexed as “ γ_C ” carbon expanded austenite (200). A second expanded austenite peak (111) appears for $2\theta = 37.71^\circ$. Analysis of the diffraction peaks gives an average expanded austenite lattice parameter of 0.4107 nm. Compared to the results for carbon expanded austenite for the 316L ASS, 0.369 nm such lattice parameter is much higher and high carbon supersaturation would be expected. For F51 DSS relative expansion of austenite lattice parameter (γ_C) when compared to the untreated austenite ($\Delta a/a$) was calculated as 14.4%.

The enrichment of the stabilizing austenite element (C) is responsible for the transformation of ferrite into austenite in the carburized case. Such behaviour was described by Christiansen and Somers (2005) after nitriding an AISI 329 duplex stainless steel: austenite and ferrite transformed to expanded austenite in the nitrated case, the austenite and ferrite original peaks being substituted by a broader one of expanded austenite. The low temperature carburizing phase transformation sequence can be described by ferrite first transforming to austenite followed by austenite transformation to expanded austenite (γ_C) (Christiansen and Somers, 2005, Bell, 2008, Larisch, et al., 1999). This phase transformation would be assisted by high nickel contents present in duplex stainless steels (Larisch, et al., 1999) such as the AISI F51. Berns and Kübl (2004) and Garzón and Tschiptschin (2006) found ferrite to austenite transformation after high temperature gas nitriding duplex stainless steel, as well.

A low intensity peak for $2\theta = 64.31^\circ$ was observed and indexed as ferrite (200) with a 0.290 nm lattice parameter. When comparing the lattice expansion to that of the untreated ferrite 0.286 nm, one can see that the difference is less than 1.4% and not enough to be clearly identified as expanded ferrite. This peak is probably originated from diffraction of the X-ray field in the ferrite grains underneath the carburized case, and might be influenced by high compressive stress fields built up on the formation of expanded austenite layer. Such behavior must be best studied and understood in future works.

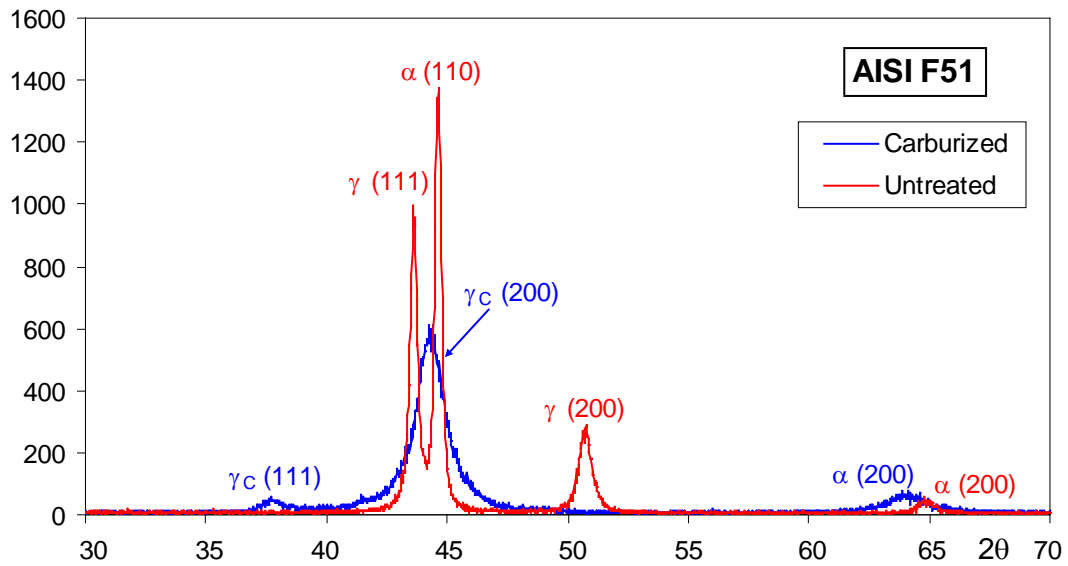


Figure 3 – XRD diffraction patterns for AISI F51 steel before and after carburization.

Low temperature plasma carburizing of AISI F51 DSS leads, also, to strong surface hardening, as a consequence of the residual stresses associated to the carbon induced expansion of the FCC lattice. However, the hardening effect is much higher than that observed for the 316L ASS. Comparing the lattice expansion of the carbon expanded austenite layer observed for both steels, ($\Delta a/a$) for the F51 DSS 14.4% and ($\Delta a/a$) for the 316L ASS only 2.8%.

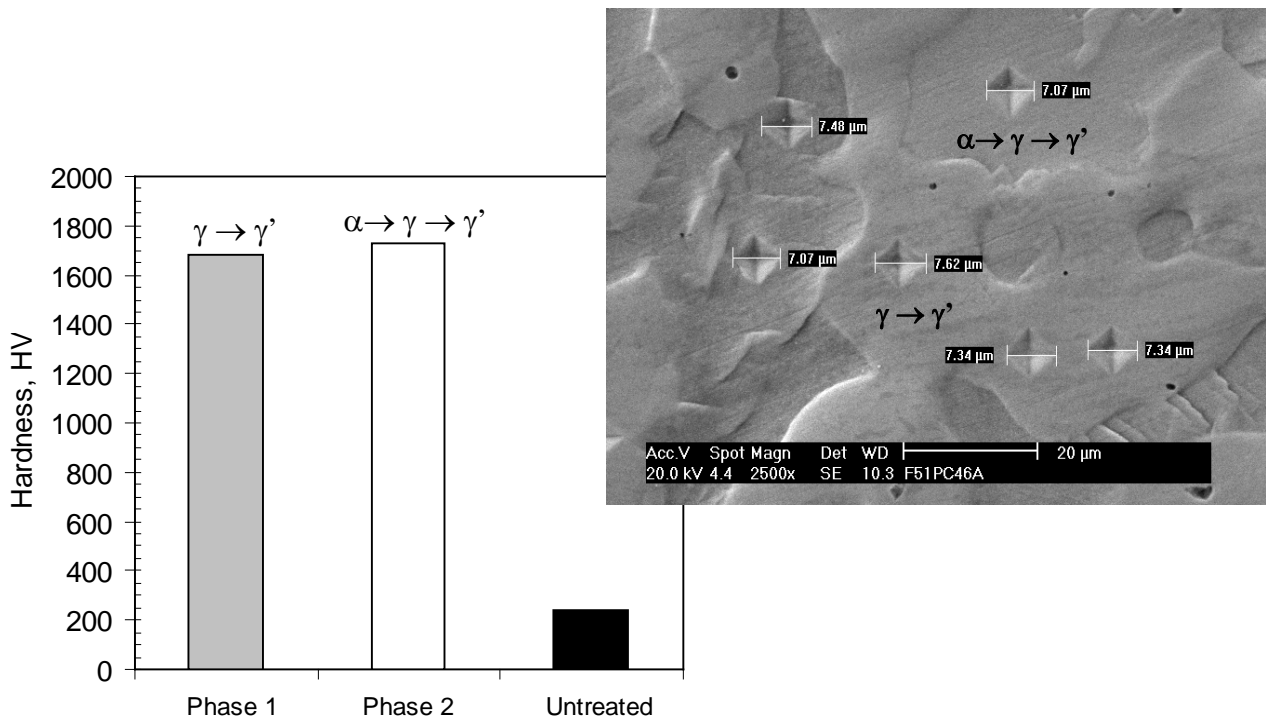


Figure 4 – Surface hardness after low temperature plasma carburizing AISI F51 DSS and SEM image showing measurements of Vickers indentations.

The lattice distortion is a consequence of a carbon colossal supersaturation [9], which leads to very high compressive stresses, increasing the surface hardness up to 1700 HV0.05, almost seven times higher than the untreated state, 240 HV0.05.

4. Conclusions

Low temperature plasma carburizing was effective in changing the surface microstructure and hardness of AISI 316L austenitic and AISI F51 duplex stainless steels.

- For the 316L ASS carbon supersaturation is responsible for the formation of a carbon rich expanded austenite (γ_C) layer. High compressive residual stresses contribute to the increase of surface hardness up to 1010 HV 0.05, five times the hardness of the untreated substrate.
- For the AISI F51 DSS the starting austenite-ferrite microstructure gets supersaturated in carbon leading to the formation of carbon expanded austenite (γ_C) as the unique phase. Carbon expanded austenite is formed not only by the lattice expansion of pre-existing austenite, but preceded by transformation of the pre-existing ferrite grains to austenite followed by formation of expanded austenite. As a consequence of the austenite lattice expansion, up to 0.4107 nm, high compressive residual stresses are present in the carburized case resulting in a strong hardening effect. This phenomenon is responsible for the intense surface hardening up to 1700HV0.05.

5. Acknowledgements

To the São Paulo State Research Foundation, FAPESP, for the financial support to this research. 2003/10157-2.

6. References

- BELL, T. Current status of supersaturated surface engineered S-phase materials, *Key Engineering Materials*, v. 373-374, p. 289-295, 2008.
- BERNS, H., KÜBL, A. Reduction in wear of sewage pump through solution nitriding, *Wear*, v. 256, p. 16-20, 2004.
- BORGIOLI, F., FOSSATI, A., GALVANETTO, E., BACCI, T. Glow-discharge nitriding of AISI 316L austenitic stainless steel: influence of treatment temperature, *Surface and Coatings Technology*, v. 200, p. 2474-2480, 2005.
- CAO; Y., ERNST; F., MICHAL; G.M. Colossal carbon supersaturation in austenitic stainless steels carburized at low temperature, *Acta Materialia*, v. 51, p. 4171-4181, 2003.
- CAVALIERE, P., ZAVARISE, G., PERILLO, M. Modeling Carburizing and Nitriding Processes *Computational Materials Science*, v. 46, p. 26-35, 2009.
- CESCHINI; L., MINAK, G. Fatigue behaviour of low temperature carburized AISI 316L austenitic stainless steel, *Surface and Coating Technology*, v. 202, p. 1778-1784, 2008.
- CHRISTIANSEN and SOMERS; M.A.J. Low temperature gaseous nitriding and carburizing of stainless steels, *Surface Engineering*, v. 21, p. 445-455, 2005.
- CZERWIEC, T., RENEVIER, N., MICHEL, H. Low-temperature plasma-assisted nitriding, *Surface and Coatings Technology*, v. 131, p. 267-277, 2000.
- Fewell, M.P., MICHELL, D.R.G., PRIEST, J.M., SHORT, K.T., COLLINS, G.A. The nature of expanded austenite, *Surface and Coatings Technology*, v.131, p. 300-306, 2000.
- GARZON, C.M., TSCHIPTSCHIN, A.P. EBSD texture analysis of a high temperature gas nitrided duplex stainless steel, *Materials Science and Engineering A*, v. 411, p. 230-238, 2006.
- International Centre for Diffraction Data (ICDD), H.J. Goldschmidt, *Metallurgia*, 40 (1949) 103.
- International Centre for Diffraction Data (ICDD), H.E. Swanson et al., *Nat. Bur. Stand (U.S.), Circ.* 509, 4, 3 (1955).
- LARISCH; B., BRUSKY; U., SPIED, H.-J. Plasma nitriding stainless steels at low temperature, *Surface and Coatings Technology*, v. 116-119, p. 205-211, 1999.
- LIANG, W., BIN, X., ZHIWEI, Y., YAQIN, S. The wear and corrosion properties of stainless steel nitrided by low-pressure plasma-arc source ion nitriding at low temperatures, *Surface and Coatings Technology*, v. 130, p. 304-308, 2000.
- MICHAL; G.M., ERNST; F., KAHN; H., CAO; Y., AGARWAL; N., HEUER, A.H. Carbon supersaturation due to paraequilibrium carburization: Stainless steels with greatly improved mechanical properties, *Acta Materialia*, v. 54, p. 1597-1606, 2006.
- MINGOLO, N., PINEDO, C.E., TSCHIPTSCHIN, A.P. On the formation of expanded austenite during plasma nitriding of an AISI 316L austenitic stainless steel, *Surface and Coatings Technology*, v. 201, p. 4215-4218, 2006.
- RENEVIER, N., COLLIGNON, P., MICHEL H., CZERWIEC, T. Low temperature plasma nitriding of AISI 316L austenitic stainless steel and Ti in a low pressure arc discharge, *Surface and Coatings Technology*, v. 111, p. 128-133, 1999.
- SOUZA, R.M., IGNAT, M., PINEDO, C.E., TSCHIPTSCHIN, A.P. Structure and properties of low temperature plasma carburized austenitic stainless steel, *Surface and Coatings Technology*, v. 204, p. 1102-1105, 2009.
- SUN; Y., LI; X., BELL; T. Low temperature plasma carburizing of austenitic stainless steels for improved wear and corrosion resistance, *Surface Engineering*, v. 15, p. 49-54, 1999.

Corresponding author: Carlos Eduardo Pinedo (pinedo@heattech.com.br)